

Fourth Quarterly Progress Report  
March 9 - June 9, 1966

A STUDY OF THE ADHESION AND COHESION OF METALS

By:

M. J. Hordon

June, 1966

Approved by:

*Norman Beecher*  
Norman Beecher  
Assistant Director  
of Research

Reviewed by:

*Frank J. Salomone*  
Frank J. Salomone  
Contracts Manager

Prepared Under Contract No. NASw-1168  
by:

Norton Exploratory Research Division  
National Research Corporation  
Cambridge, Massachusetts 02142

For

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

FACILITY FORM 802

N66 32795	(THRU)
(ACCESSION NUMBER)	3
16	(CODE)
(PAGES)	17
CR-76920	(CATEGORY)
(NASA CR OR TMX OR AD NUMBER)	

## TABLE OF CONTENTS

	PAGE
SUMMARY	1
INTRODUCTION	1
APPARATUS	2
Vacuum System	3
Specimen Selector Device	3
Abrasion Device	4
ADHESION SPECIMENS	6
EXPERIMENTAL PROGRESS	9
FUTURE WORK	10

# A STUDY OF THE ADHESION AND COHESION OF METALS

By M. J. Hordon  
National Research Corporation

## SUMMARY

*N66-32795*

During the first quarter of the adhesion program extension, modifications and improvements to the test apparatus were made including the redesign of the refrigeration and cooling system and the specimen selector and abrader mechanisms. The modified apparatus components were installed and preliminary vacuum pumping operations were begun.

Selected specimens with various lattice structures were prepared for adhesion testing. Heat treatment procedures to produce nearly equivalent yield strengths were carried out. Adhesion testing of Cu, Ti, Ni, Ta, Co, beta-brass, 430 and 304 grade stainless steels and alpha and zeta phase Ag-Sn alloys are scheduled for the succeeding period.

*Author*

## INTRODUCTION

In the current program, NRC is continuing an investigation of various aspects of solid adhesion behavior in order to acquire a deeper understanding of the fundamentals of the bonding mechanism. During the last quarter, an interim summary report was prepared covering the work carried out at NRC in the course of the initial twelve months investigation. This work included studies of contact surface cleanliness and micro-contour on the degree of adhesion. In addition, a substantial body of data was generated on the effects of properties such as yield strength and solid solubility on the degree of bonding.

In the current program extension, it is planned to investigate the effect of lattice structure and crystallographic orientation on the adhesion bond strength. It has been postulated that the ease of metallic bonding is directly proportional to the density of atomic bonds across the weld interface. Hence, metals with face centered cubic (FCC) lattices such as Cu and Ni with bond coordination numbers of 12 should exhibit greater bond strengths than metals with lower coordination lattices such as body centered cubic (BCC) or close packed hexagonal (HCP) structures.

Moreover, for each lattice type, the concentration of bonds per surface atomic site will vary with planar orientation. For cubic metals, the (111) plane surface is the plane of maximum atomic density and minimum interatomic distance. It is likely, therefore, that atomic misorientation or bond distortion will be minimized with a (111) crystal interface and microscopic adhesion will be enhanced.

To investigate these effects, samples of various metal lattice structures as well as single crystal specimens of copper of varying contact surface orientation were planned for adhesion testing. In order to accommodate the new specimens, various modifications to the adhesion test apparatus were considered necessary. During the last quarter, sample preparation and equipment modification were undertaken. Comparative adhesion tests of varying lattice structure are scheduled for the second program quarter.

## APPARATUS

The adhesion testing vacuum apparatus scheduled for use in the present work has been extensively described in prior progress reports and in the Interim Summary Report for NASA Contract No. NASw-1168. The apparatus consists, essentially, of a 2000 pound load capacity compression cage positioned in an ultra-high vacuum chamber capable of pressures below  $7 \times 10^{-10}$  with full testing assembly. In addition, the two loading stages are designed to hold up to eight specimens each so that any one of 64 metal pair combinations may be tested in one pumping operation.

The vacuum chamber also contains ports for ion beam bombardment of both specimen surfaces, a feed-through port for a vibratory or rotary wire brush abrasion device and manipulative devices for positioning the loading stages to bring the desired specimens into contact.

In the course of adhesion testing experience, it became apparent that several design modifications would be extremely useful in improving the performance of the apparatus. In particular, three areas that required redesign were; (1) modifications and repair of the general vacuum system and associated refrigeration and cooling system, (2) redesign of the specimen selector mechanisms necessary for rapid rotation of the loading stages and (3) modification of the wire brush abrasion device. The necessary equipment alterations were scheduled for the initial quarter of the extended program.

### Vacuum System

The vacuum system consists of a six-inch NRC diffusion pump in series with a two-inch diffusion pump and a mechanical pump. The gas exhaust outlet is provided with two liquid nitrogen traps as well as a refrigerated right angle trap. The lower section of the test chamber and the evacuation port are cooled to  $-35^{\circ}\text{F}$  by circulating a refrigerant, Lexol, through copper cooling coils.

The apparatus contains two major access flanges which contain double "O"-ring seals. Previously water cooled, the "O"-ring seals constituted an important restriction in the limiting residual pressure obtained after thermal outgassing. In order to further reduce outgassing of the "O"-rings, copper coils were placed in the water channels so that the cold refrigerant could be utilized to cool the flanges. In this way, the flange seals can be brought to about  $-35^{\circ}\text{F}$  during a pumping operation and gas emission from the organic rings substantially reduced.

In addition to changes in the cooling system, changes in the placement of instrument ports on the test chamber were made to reduce the number of potential leaks. The modifications described above have been substantially completed and the vacuum system is ready for operation.

### Specimen Selector Device

In order to select the pair of specimen surfaces for adhesion testing, the loading stage wheel must be rotated in the vacuum so that the proper mating surfaces are in the vertical position. In the case of the inert gas ion bombardment surface

cleaning procedure, the specimen pair are initially placed in a horizontal position during the bombardment period and then must be rapidly rotated to the vertical loading position. It is understood that the rotational axes of the two loading stages are at right angles to each other in order to accomodate two ion guns.

Considerable design effort has been directed toward the problem of specimen selection and indexing. In the initial design, the loading stages were rotated by two manipulative arms releasing a spring pin so that the stages could travel through 1/8 of a revolution and lock the next specimen in the proper loading position. The time period, however, to travel through 90° rotation, as required for ion bombardment cleaning, was in excess of 60 sec. for each loading stage.

During the last year, a new rapid action specimen selector mechanism was designed based on a weight and pulley arrangement. In this mode, the desired specimens could be quickly rotated through 90° in less than 10 sec. However, the pulley action necessitated severe limitations on the flexibility of adhesion testing since the total amount of rotation was restricted to about two revolutions in one direction.

In the last quarter, this important component mechanism has been redesigned to provide the necessary rapidity and flexibility. In place of the weight and pulley action together with the manipulative release arms, specially designed vacuum sealed rotators have been mounted on the test chamber so that the loading stages can be rotated at will from outside the chamber. A detail of the rotator mechanism is shown in Fig. 1. Using a copper gasket as the vacuum seal, rotational motion is applied by means of an eccentric cam within a flexible metal bellows to the loading stages. The rotator can apply torques up to 5 in-lbs through a distance of 12 in. through the vacuum chamber wall.

#### Abrasion Device

In previous adhesion tests, contact surfaces have been successfully cleaned of oxide films by means of an abrasive mechanism such as a wire brush. Using a small vacuum sealed D. C. motor element, the mating surfaces were abraded immediately before contact. However, difficulties were encountered due to outgassing of the motor assemblies within the vacuum and to heating restrictions. Frequently, the motor bearings seized in the high vacuum environment.

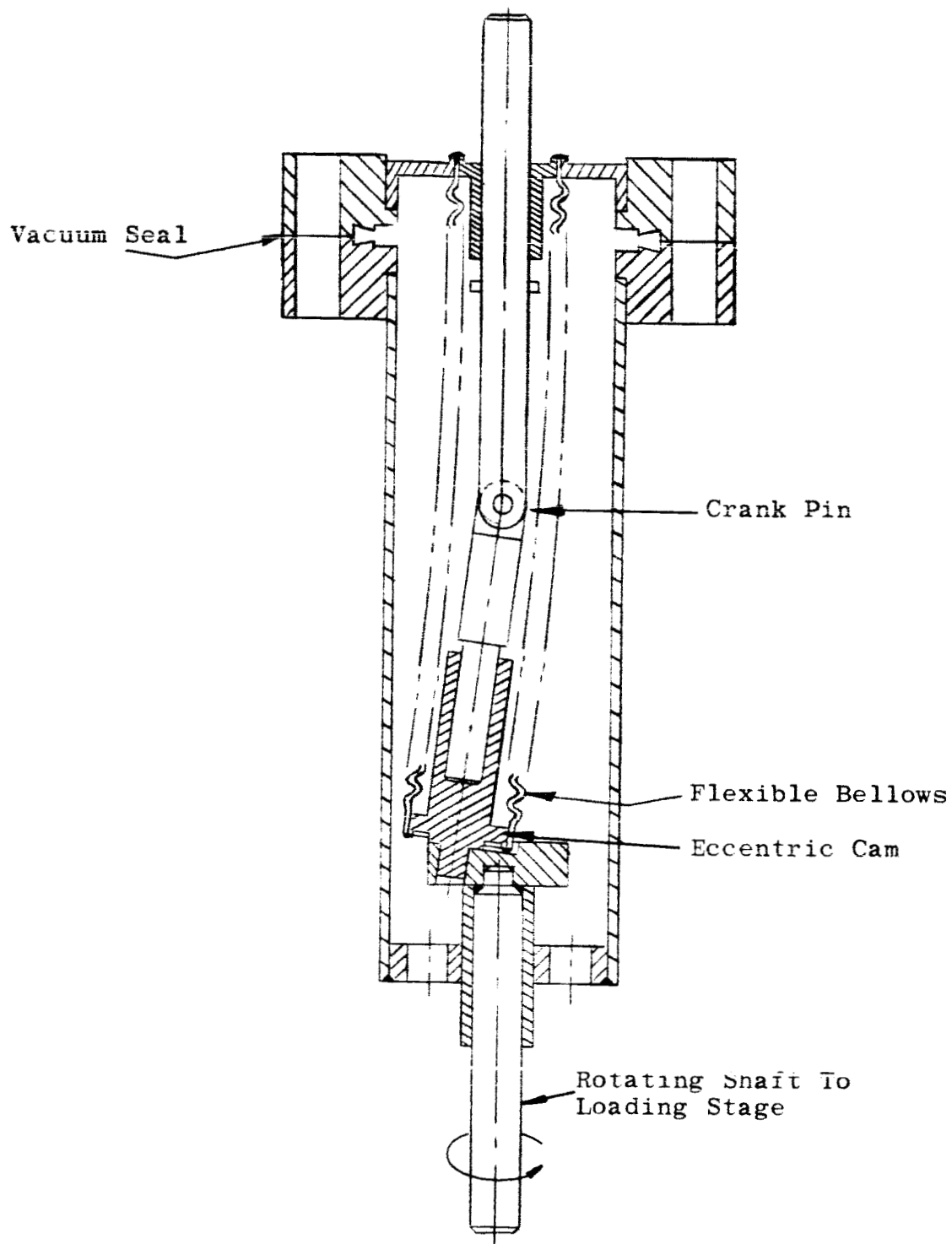


Fig. 1

Specimen Selector Rotator Device

To avoid these difficulties, the abrasion mechanism was re-designed to position the motor drive outside the vacuum chamber. A detail of the device is shown in Fig. 2. In this design, the motor drive actuates a vibratory abrader through a flexible metal bellows. The vibration amplitude is about 3/8 inch and is powered by a 1/15 H. P. A. C. motor capable of vibration frequencies up to 6000 cycles/min. The abrader consists of a stainless steel wire brush flattened on two opposite sides in order to obtain good contact with both adhesion surfaces.

With this design, the motor drive is removed from the vacuum chamber and gas leakage from the motor seals as well as limitations to the thermal outgassing temperature are avoided.

#### ADHESION SPECIMENS

In order to obtain comparative adhesion data with changes in lattice structure, the metals and alloys listed in Table I were prepared for self-adhesion tests. During the initial quarter, these materials were acquired and prepared for heat treatment prior to testing. The heat treating procedure for each sample type is also given in Table I.

The materials selected for adhesion analysis can be divided into two major groups depending on chemical composition. In Group A are the pure elemental metals listed below in two sub-groups:

Cu	Ni
Ti	Ta
	Co

which can be matched reasonably closely within each subgroup with respect to mechanical properties such as the elastic modulus and the compressive yield stress. The object in selecting these materials for bonding comparison was to examine the effect of differing lattice structure on the adhesion properties in the absence of pronounced variations in mechanical behavior which would obscure the influence of structure.

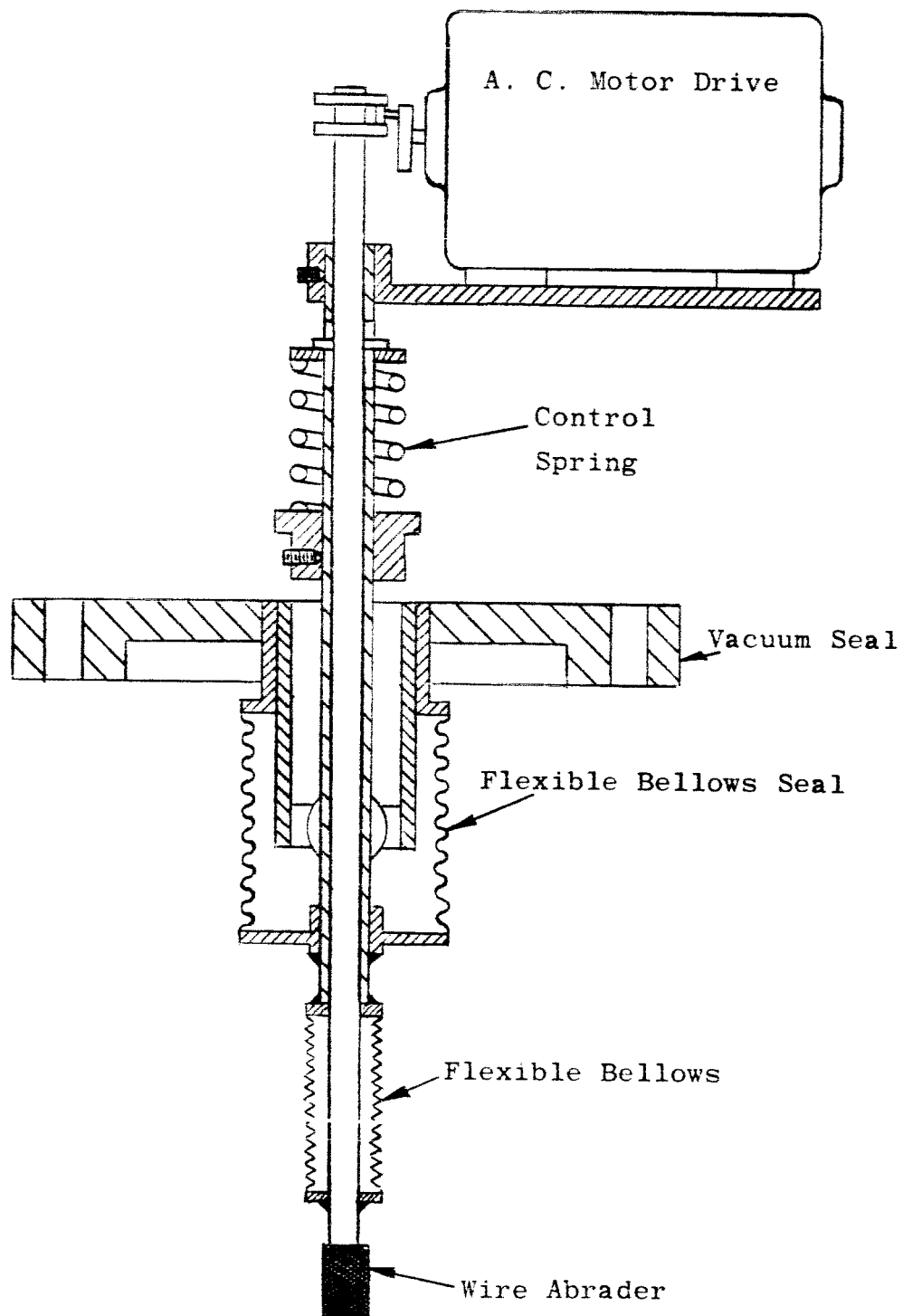


Fig. 2

Vibratory Abrasion Device

TABLE I  
MATERIALS FOR ADHESION SPECIMENS

Metal	Structure	Elastic Modulus E (psi x 10 <sup>-6</sup> )	Heat Treatment
Group A			
Cu	FCC	16.0	800°C, 1 hr.
Ti	HCP	16.8	700°C, 2 hrs.
Ni	FCC	30	900°C, 1 hr.
Ta	BCC	27	1200°C, 1 hr.
Co	HCP	30	1200°C, 2 hrs.
Group B			
304 S. S.	FCC	30	1050°C, 1 hr.
430 S. S.	BCC	30	850°C, 1 hr.
Beta-Brass (61 Cu-39 Zn)	FCC	15	750°C, 1 hr. (slow cool)
" "	BCT (Distort)	15	850°C, 1 hr. (quench into iced brine)
93 Ag - 7 Sn	FCC	10	550°C, 1 hr.
84 Ag - 16 Sn	HCP	10	600°C, 1 hr.

In Group B are alloys in which the lattice structure can be varied either by small changes in the composition or by thermal treatment. For the two stainless steel alloys, the addition of about 10% Ni essentially transforms a ferritic BCC structure into an austenitic FCC structure. In particular, the ferritic 430 grade stainless has a largely similar composition to the austenitic 304 grade with the exception that the 430 grade contains no nickel. In the tin-silver alloys, a change in tin content from 7 to 14% will result in a structural phase change from FCC to HCP. In both sets of lattice transformations, the phase changes also involve changes in mechanical properties such as ductility and yield strength, but these variations can be minimized by suitable heat treatment.

In the case of the beta-brass alloy, the structure can be varied without changing the composition since the alloy undergoes an allotropic phase change upon heating above 458°C. Under severe quenching conditions, a metastable beta' (BCT) distorted cubic phase can be retained at room temperature in place of the equilibrium alpha (FCC) phase.

#### EXPERIMENTAL PROGRESS

During the initial quarter of the extended program, the principal experimental effort has been concerned with modifications and improvements to the vacuum adhesion testing system and with the preparation of specimens. The necessary changes in the refrigeration, rotary specimen selector and abrader components have been largely completed.

Preliminary vacuum operations have shown that the system may now be outgassed at temperatures in excess of 300°C, substantially reducing the residual gas load with indicated total pressures in the  $5 \times 10^{-10}$  Torr range at room temperature. The increase in vacuum capability of the adhesion test system may be attributed to lower flange seal temperatures, removal of the abrader motor from the vacuum chamber and the elimination of several excess components which contributed to the gas load.

In the same period, samples of copper, titanium, nickel, tantalum, 430 Grade and 304 Grade stainless steels have been machined from 3/4 inch diameter wrought bar stock. Cobalt samples were machined from 3/4 inch diameter wrought and extruded rod. The beta-brass alloy samples were machined from 1/2 inch thick plate. Samples approximately 1/2 x 1/2 x 3/8 inch in size were rough cut for heat treating prior to final machining. In addition, samples approximately 1 inch in height x 3/4 inch in diameter were prepared for testing of the compressive yield stress after heat treatment.

Specimens of alpha and zeta phase silver-tin alloys were prepared in the laboratory by vacuum melting and casting at 1050°C. In this procedure, the required charges of pure silver and tin were placed into a platinum wire resistance vacuum furnace and quickly brought to temperature at about  $3 \times 10^{-5}$  torr. After agitation of the melt to remove surface dross, the melts were cast into carbon molds about 1 inch in diameter. Castings up to 4 inches in length and weighing about 280 grams were obtained.

Subsequent chemical analysis of the castings showed that the alpha Ag-Sn alloy had a tin content of 7.22% well within the solid solubility limit of about 10% for the alpha phase at room temperature. The zeta phase alloy had a tin concentration of 16.40%, within the phase solubility limits of 12.8% and 19.5% at room temperature. After analysis, the castings were sectioned to produce specimen blanks about 1/2 x 1/2 x 3/8 inches in size for annealing treatments and yield strength determinations.

#### FUTURE WORK

In the current quarter of the adhesion program, preliminary tests are scheduled with the modified apparatus. After qualification, a series of adhesion tests are scheduled at room temperature for samples of differing lattice structure but equivalent yield coefficient. The yield coefficient is defined as the ratio of the applied contact stress to the compressive yield stress.

Sample geometry will be tailored so that the contact area will produce the required stresses at the maximum applied load. Specimen surfaces will be cleaned prior to testing by vibratory wire brush abrasion.